

compared to an uncoated, bare capillary tube. FIG. 9 illustrates separation results, the solid line indicating separation on a multilayer prepared as described above, and the dashed line indicating the result with the uncoated capillary. Separation conditions were as follows: 37 cm capillary length, with 3.0 cm to the sensor, 50  $\mu$ m internal diameter, 20 mM phosphate at pH 4.0 as running electrolyte, 5 kV operating voltage, electrokinetic injection of the sample, and detection by the sensor at 254 nm. FIG. 9 shows separation of peaks for 1) acetone, and 2) phenol. The results indicate that, while there was no separation in the bare silica capillary, there was significant separation evident using the multilayer.

FIG. 10 illustrates separation peaks for 1) acetone, 2) fluorobenzene, 3) phenol, and 4) p-cresol on a multilayer deposited as described, with the modification of all layers being deposited in the presence of 0.5 M NaCl and 30% ethanol. Separation conditions were as follows: 37 cm capillary length, with 30 cm to the sensor, 50  $\mu$ m internal diameter, 20 mM phosphate at pH 4.0 as running electrolyte, 15 kV operating voltage, electrokinetic injection of the sample, and detection by the sensor at 254 nm.

The multilayer formed in the presence of ethanol did not appear to allow elution of a large neutral solute such as naphthalene. Presence of ethanol during multilayer deposition was shown to produce thicker multilayers, and it is theorized that a large neutral solute would tend to be retained within the multilayer. Therefore, a multilayer formed in the presence of 0.5 M NaCl and 20% acetonitrile was tested for separation and elution of naphthalene. Acetonitrile was used at a lower concentration and, since it is a slightly more polar molecule than ethanol, it was expected to form a thinner multilayer, therefore having a smaller tendency to retain large neutral molecules. FIG. 11 shows separation of peaks of 1) acetone, and 2) naphthalene. Separation conditions included: 80% 20 mM phosphate/20% acetonitrile as the running electrolyte and mobile phase; pH 6.0, 37 cm capillary length with 30 cm to the sensor, 50  $\mu$ m internal diameter, 15 kV applied voltage, electrokinetic sample injection at 5 kV for 5 seconds, and detection at 254 nm.

#### Example 4

##### Separation Using Multilayer Coated Particles

A capillary coated with a polyelectrolyte multilayer was prepared as previously described. Polyelectrolyte multilayer coated particles were prepared also as described above, and packed into the capillary, as illustrated in FIG. 3.

Capillary electrochromatography was performed using water as the mobile phase. Acetone was used as the void time marker. Phenol and naphthol were used as test compounds for the separation. Separation conditions were as described in Example 3, above. Illustrative separation results for phenol and acetone are shown in FIG. 12.

In the drawings and specification, there have been disclosed a preferred embodiment of the invention, and although specific terms are employed, the terms are used in a descriptive sense only and not for purposes of limitation. The invention has been described in considerable detail with specific reference to these illustrated embodiments. It will be apparent, however, that various modifications and changes can be made within the spirit and scope of the invention.

What is claimed is:

1. A capillary tube for analytical separations of macromolecules, said capillary tube comprising:

a passage defined by capillary walls comprising fused silica; and

a multilayer positioned within said passage adjacent said walls, wherein said multilayer comprises a plurality of polyelectrolyte layers; and

a plurality of particles positioned within said passage, wherein each individual particle comprises a multilayer including a plurality of polyelectrolyte layers.

2. The capillary tube of claim 1, wherein said particles comprise fused silica.

3. The capillary tube of claim 1, wherein said passage comprises a substantially cylindrical void space having a diameter of from about five micrometers to about one hundred micrometers.

4. The capillary tube of claim 1, wherein said capillary tube has a first end, a second end, and a lengthwise dimension extending therebetween, and wherein said passage extends along the lengthwise dimension from a first opening positioned at said first end to a second opening positioned at said second end.

5. The capillary tube of claim 1, wherein said multilayer further comprises a plurality of layers of an organic cationic polyelectrolyte.

6. The capillary tube of claim 1, wherein said multilayer further comprises a plurality of layers of an organic anionic polyelectrolyte.

7. The capillary tube of claim 1, wherein said multilayer further comprises alternating of layers of an organic cationic polyelectrolyte and an organic anionic polyelectrolyte.

8. The capillary tube of claim 1, wherein concentration of said polyelectrolyte varies within the multilayer.

9. The capillary tube of claim 1, wherein said multilayer is deposited in the presence of sodium chloride.

10. The capillary tube of claim 1, wherein said multilayer comprises sodium chloride.

11. The capillary tube of claim 1, wherein said multilayer comprises a modifier selected from organic solvents.

12. The capillary tube of claim 1, wherein said modifier comprises ethanol.

13. The capillary tube of claim 1, wherein said multilayer comprises a weak polyelectrolyte.

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